

YAGUPOL'SKIY, L.M.; VISHNEVSKAYA, G.O.; YAVORSKIY, D.F.; GRUZ, B.Ye.;  
MAKSIMENKO, A.S.; KHASKIN, I.G.; GONSETSKAYA, Ya.V.; KIPRIANOV,  
A.I.

Improvement in the method for producing p-nitrophenylchloro-  
methylcarbinole. Med.prom. 13 no.3:20-21 Mr '59.

(MIRA 12:5)

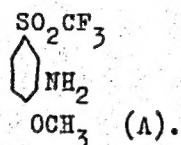
1. Institut organicheskoy khimii AN USSR i Kiyevskiy khimiko-  
farmatsevticheskiy zavod imeni M.V.Lomonosova.  
(METHANOL)

AUTHORS: Yagupol'skiy, L. M., Marenets, M. S. SOV/79-29-1-58/74

TITLE: Synthesis of the Derivatives of Phenyl Trifluoro Methyl Sulfone (Sintez proizvodnykh feniltriftormetilsul'fona)

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 1, pp 278 - 283 (USSR)

ABSTRACT: It was the aim of the present study to prepare the intermediate products for the synthesis of azo- and cyanine dyes which contain a trifluoro methyl sulfonyl group. 3-amino-4-fluoro- and 3-amino-4-chloro-phenyl trifluoro methyl sulfones were obtained according to the mentioned scheme 1. From 3-nitro-4-chloro-phenyl trifluoro methyl sulfone by means of sodium methylate 3-nitro-4-methoxy phenyl trifluoro methyl sulfone was produced which was reduced to the amine



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Apart from this the following substances were synthesized:

Synthesis of the Derivatives of Phenyl Trifluoro Methyl Sulfone

SOV/79-29-1-58/74

2-methyl-6-trifluoro methyl sulfonyl benzimidazole, 2-5-trifluoro methyl sulfonyl benzthiazole according to scheme 2. The latter base was transformed into the quaternary salt from which carboxy anine and the dye styril were obtained. The group  $\text{CF}_3\text{SO}_2$  in position 5 of the benzthiazole nucleus as well as other electronegative substituents in the same position ( $\text{NO}_2$ ,  $\text{CF}_3$ , Refs 1,2) almost does not change the absorption maximum of thiacyanocyanine. In the dye styril the group  $\text{CF}_3\text{SO}_2$  shifts the absorption maximum to 25 mμ towards the side of long waves (Table). 3-nitro-4-hydrazine phenyl trifluoro methyl sulfone was also obtained. There are 1 table and 9 references, 5 of which are Soviet. Institut organicheskoy khimii Akademii nauk Ukrainskoy SSR (Institute of Organic Chemistry of the Academy of Sciences, Ukr SSR) November 25, 1957

ASSOCIATION:

SUBMITTED:

Card 2/2

AUTHORS:

Yagupol'skiy, L. M., Troitskaya, V. I. SOV/79-29-2-42/71

TITLE:

Fluor-containing Trichloro-phosphazo-sulfonaryls and Their Derivatives (Ftorsoderzhashchiye trikhlorfosfazosul'fonarily i ikh proizvodnyye)

PERIODICAL:

Zhurnal obshchey khimii, 1959, Vol 29, Nr 2, pp 552-556 (USSR)

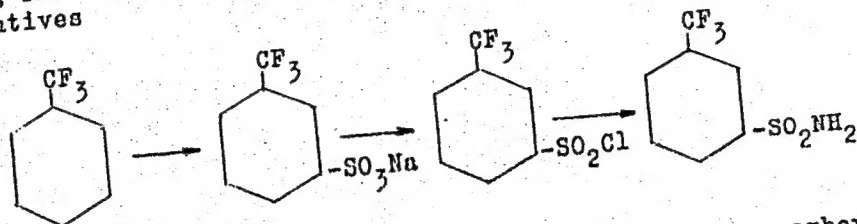
ABSTRACT:

At present several active insecticides with fluorine atoms in the molecule (Ref 1) are known. Preparations, simultaneously containing fluorine and phosphorus atoms are of special interest. The synthesis and investigation of trichloro-phosphazo-sulfonaryls and their derivatives for such insecticides as contain fluorine atoms or trifluoro-methyl groups as substituents in the aromatic nucleus are the aim of the present article. For this purpose, the n-fluoro-benzene-sulfamide (Ref 3) and the hitherto unknown m-trifluoro-methyl-phenyl-sulfamide were synthesized. The latter was obtained according to the scheme

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Fluor-containing Trichloro-phosphazo-sulfonaryls  
and Their Derivatives

SOV/79-29-2-42/71



Its structure was proved by transformation into m-carboxy-benzene-sulfamide (Ref 4) with sulfuric acid. By reaction with  $\text{PCl}_5$  the sulfamides thus obtained yield trichloro-phosphazo-sulfonaryls (Ref 5):  

$$\text{Ar-SO}_2\text{NH}_2 + \text{PCl}_5 \rightarrow \text{Ar-SO}_2\text{N=PCl}_3$$
 where  $\text{Ar} = m\text{-FC}_6\text{H}_4$  (I), and  $m\text{-CF}_3\text{C}_6\text{H}_4$  (II). From (I) and (II) the dichloro anhydrides and the corresponding phenyl-sulfonamide-phosphoric acids were obtained by hydrolysis and acidolysis. Also the monochloro anhydrid of fluoro-phenyl-sulfonamide-phosphoric acid (Scheme 3) was separated. With alcoholates and phenolates the compounds (I) and (II) condense to ether (Tables 1,2).

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Fluor-containing Trichloro-phosphazo-sulfonaryls  
and Their Derivatives

SOV/79-29-2-42/71

The esters of n-fluoro- and m-trifluoro-methyl-sulfonamide-phosphoric acids are colorless compounds of crystalline nature. Applied as insecticides the preparations 1 and 7 specified in table 1 exhibit little activity. There are 2 tables and 5 references, 2 of which are Soviet.

ASSOCIATION:

Institut organicheskoy khimii Akademii nauk USSR (Institute of Organic Chemistry of the Academy of Sciences, UkrSSR)

SUBMITTED:

January 8, 1958

Card 3/3



SOV/79-29-4-58/77

5(3)

AUTHOR:

Yagupol'skiy, L. M.

TITLE:

Cyanin Dyes Containing Fluorine (Tsianinovyye krasiteli, sodershashchiye ftor). VI. Synthesis of Cyanin Dyes From 5-Trifluoromethoxy-6-acetylaminothiazole (VI. Sintez tsianinovykh krasiteley iz 5-triftormetoksi-6-atsetilaminobenzthiazola)

PERIODICAL:

Zhurnal obshchey khimii, 1959, Vol 29, Nr 4, pp 1312-1315 (USSR)

ABSTRACT:

In the previous paper (Ref 1) the synthesis of thiocarbocyanins containing trifluoromethoxy groups as substituents in positions 5 and 6 of the benzothiazole nucleus was described. The introduction of methoxy groups into the 5,5'-positions of 6,6'-disubstituted thiocarbocyanins is known to cause an additive shift of absorption maximum of the dyes toward the long-wave range of the spectrum. It was interesting to find out in what way the trifluoromethoxy groups introduced into positions 5,5' would influence the absorption maxima of 6,6'-disubstituted thiocarbocyanins. The initial material used for the synthesis of these dyes was the ether (I), which was obtained from a mixture of the relevant nitroacetylamino derivatives (Ref 1). The structure of this ether was proved by removing the acetyl from it and re-

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SOV/79-29-4-58/77

## Cyanin Dyes Containing Fluorine. VI. Synthesis of Cyanin Dyes From 5-Trifluoromethoxy-6-acetylaninobenzothiazole

ducing it to the diamine which, like the other paradiamines, exhibited an intense indamine reaction with iron trichloride. Like the corresponding methoxy derivative (Ref 3) it was easily oxidized to the p-quinone (Scheme 1). Compound (I) was transformed into a benzothiazole (IV) according to reference 1 (Scheme 2). By diazotizing the amine (IV) and substituting other groups new bases were obtained, viz. 6-chloro-, 6-iodo-, and 6-methylmercapto-5-trifluoromethoxy-2-methylbenzothiazoles, which, in turn, were transformed into the quaternary salts and thiocarbocyanins. The absorption maxima of the synthesized cyanin dyes in alcohol are given in the table where for comparative purposes, the absorption maxima of the corresponding dyes without trifluoromethoxy groups are also given. Therefrom it may be concluded that the trifluoromethoxy group in the 5,5'-positions of the benzothiazole nucleus has a slight effect only on the absorption maxima of the dyes containing weakly electropositive substituents in the 6,6'-positions. There are 1 table and 3 Soviet references.

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SOV/79-29-4-58/77

Cyanin Dyes Containing Fluorine. VI. Synthesis of Cyanin Dyes From 5-Trifluoromethoxy-6-acetylaminobenzothiazole

ASSOCIATION: Institut organicheskoy khimii Akademii nauk SSSR (Institute of Organic Chemistry of the Academy of Sciences, USSR)

SUBMITTED: March 6, 1958

Card 3/3

5 (3)

AUTHORS:

Yagupol'skiy, L. M., Troitskaya, V. I.

SOV/79-29-7-68/83

TITLE:

Cyanine Dyes Containing Fluorine (Tsianinovyye krasiteli, sodershashchiye ftor). VII. Synthesis of Cyanine Dyes From 6-Trifluoro-methyl and 6-Trifluoro-methyl-sulfonyl-benzimidazole (VII. Sintez tsianinovyykh krasiteley iz 6-triftormetil- i 6-triftormetilsul'fonilbenzimidazola)

PERIODICAL:

Zhurnal obshchey khimii, 1959, Vol 29, Nr 7, pp 2409-2416 (USSR)

ABSTRACT:

The cyanine dyes, derivatives of benzimidazole, are but little investigated with some exceptions (Refs 1-5). The simplest dye of this series, the 1,1',3,3'-tetramethyl-imidocarbo cyanine iodide (formula on page 2409) was first synthesized by Ogata (Ref 1) and then by A. I. Kiprianov (Ref 2). The purpose of the present paper was the synthesis of the imidocarbo cyanines which have as substituents electrophilic trifluoro-methyl groups and trifluoro-methyl-sulfonyl groups. The necessary derivatives of benzimidazole were obtained according to scheme 1 and the quaternary salts from these bases (Formula 2). From the quaternary salts the cyanine dyes were synthesized, formula and absorption maxima of which are presented in tables 1 and 2. The symmetrical imidocarbo cyanine dyes were obtained by boiling the

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Cyanine Dyes Containing Fluorine. VII. Synthesis of SOV/79-29-7-68/83  
Cyanine Dyes From 6-Trifluoro-methyl and 6-Trifluoro-methyl-sulfonyl-  
benzimidazole

quaternary salts with orthoformic acid in nitro-benzene according to A. Van Dormael (Ref 6). The introduction of the trifluoro-methyl group and trifluoro-methyl-sulfonyl group (Ref 8) into the benzthiazole nucleus of the thiocarbo-cyanine hardly changes the absorption maximum of the dye. Table 2 gives formulae and absorption maxima of two unsymmetrical cyanine dyes and two rhodocyanines. The substitution of ethyl radicals for the methyl radicals on the nitrogen atoms of the benzimidazole nucleus causes considerable changes in the absorption maximum of the imidocarbo cyanines. The synthesis of 2-methyl-3-phenyl-6-trifluoro-methyl- and 2-methyl-3-ethyl-6-trifluoro-methyl-sulfonyl-benzimidazole was thus described. These bases, like the 2-methyl-3-phenyl-6-trifluoro-ethyl-sulfonyl-benzimidazole previously described, were then transformed into quaternary salts from which 8 symmetrical imidocarbo cyanines, 2 unsymmetrical and 2 rhodocyanines were obtained. There are 3 tables and 12 references, 6 of which are Soviet.

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Cyanine Dyes Containing Fluorine. VII. Synthesis of SOV/79-29-7-68/83  
Cyanine Dyes From 6-Trifluoro-methyl and 6-Trifluoro-methyl-sulfonyl-  
benzimidazole

ASSOCIATION: Institut organicheskoy khimii Akademii nauk Ukrainskoy SSR  
(Institute of Organic Chemistry of the Academy of Sciences of  
the Ukrainian SSR)

SUBMITTED: June 5, 1958

Card 3/3

5(3)

AUTHORS:

Yagupol'skiy, L. M., Troitskaya, V. I.

SOV/79-29-8-63/81

TITLE:

Cyanin Dyes Containing Fluorine. VIII. Synthesis of Cyanin Dyes From 4,6-Bis-(trifluoromethyl)-4-chloro-6-trifluoromethyl and 4-Chloro-6-trifluoromethylsulfonylbenzimidazole

PERIODICAL:

Zhurnal obshchey khimii, 1959, Vol 29, Nr 8, pp 2730-2736 (USSR)

ABSTRACT:

In a previous paper (Ref 1) the synthesis of imidocarbocyanins containing as substituents in position 6 of the benzimidazole nucleus trifluoromethyl- and trifluoromethylsulfonyl groups was described. In the present paper tetrasubstituted imidocarbocyanins of the general formula (A) were obtained. The benzimidazole derivatives which were used as initial products and the quaternary salts were synthesized according to the already described method (Ref 1). Table 1 gives formulas and absorption maxima of the symmetrical dyes in alcohol. By way of comparison the absorption maxima of the corresponding dyes which do not contain substituents in position 4 of the benzimidazole-nucleus are also given. As is seen from the data of table 1, the introduction of trifluoromethyl groups into positions 4,4' of 6,6-bis-(trifluoromethyl)-imido-carbocyanin (dyes I - III) causes a shift of the absorption

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Cyanin Dyes Containing Fluorine. VIII. Synthesis of SOV/79-29-8-63/81  
Cyanin Dyes From 4,6-Bis-(trifluoromethyl)-4-chloro-6-trifluoromethyl and  
4-Chloro-6-trifluoromethylsulfonylbenzimidazole

maximum towards the long waves of about 11 - 13 m $\mu$ , the introduction of the chlorine atom into the same positions a shift of only 4 m $\mu$  (dye IV) and a shift of about 3 - 6 m $\mu$  in the direction of 6,6'-bis-(trifluoromethylsulfonyl)-imidocarbocyanin (dyes V-VII). Table 2 gives formulas and absorption maxima of the 4 asymmetrical cyanin dyes. Tables 3 and 4 give the yield and melting points of symmetrical and asymmetrical dyes. There are 4 tables and 5 references, 3 of which are Soviet.

ASSOCIATION: Institut organicheskoy khimii Akademii nauk Ukrainskoy SSR  
(Institute of Organic Chemistry of the Academy of Sciences of the Ukrainskaya SSR)

SUBMITTED: July 10, 1958

Card 2/2



5(3)

SOV/79-29-9-60/76

AUTHORS: Yagupol'skiy, L. M., Fialkov, Yu. A.

TITLE: 2-Trifluoromethyl Naphthalene and Its Derivatives

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 9, pp 3082-3086 (USSR)

ABSTRACT: The trifluoromethyl derivatives of naphthalene have hitherto not been investigated. The synthesis of 1-trifluoromethyl naphthalene briefly mentioned in an American patent (Ref 1) is very insufficiently described, i.e. no constants and no exact course of synthesis, both of the final product and of 1-trichloromethyl naphthalene as initial product are given. The trichloromethyl derivatives of naphthalene are difficultly accessible (Ref 2). In the experiment made by A. N. Nesmeyanov and co-workers (Ref 3) to obtain  $\alpha$ -trichloromethyl naphthalene by thermal decomposition of the copper oxide salt of trichloroacetic acid in an excess amount of naphthalene it could not be separated. For this reason the o-chlorotrichloromethyl derivatives of naphthalene which were obtained from the corresponding oxynaphthoic acids with  $PCl_5$  were used as initial products for the synthesis of the trifluoromethyl compounds of the naphthalene series. From the three o-oxynaphthoic

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SOV/79-29-9-60/76

## 2-Trifluoromethyl Naphthalene and Its Derivatives

acids, R. Woffenstein (Ref 4) succeeded in transforming only 1-oxy-2-naphthoic acid into 1-chloro-2-trichloromethyl naphthalene; the yield is not specified. The remaining acids could be identified only in the form of the corresponding o-chloronaphthoic acids. The authors repeated Woffenstein's experiments. They succeeded in synthesizing 1-chloro-2-trichloromethyl naphthalene from 1-oxy-2-naphthoic acid in a yield of 35%. The replacement of chlorine by fluorine in 1-chloro-2-trichloromethyl naphthalene with  $\text{SbF}_3$  in the presence of  $\text{SbCl}_5$  and without the latter gave no positive results. The fluorination of 1-chloro-2-trifluoromethyl naphthalene succeeded only in solvents. In chlorobenzene a yield of 90% of 1-chloro-2-trifluoromethyl naphthalene was obtained. 1-chloro-2-trifluoromethyl naphthalene was transformed, on heating with copper cyanide in the presence of pyridine, into the nitrile of 2-trifluoromethyl-1-naphthoic acid from which the amide was obtained. This amide yielded,

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2-Trifluoromethyl Naphthalene and Its Derivatives

according to the scheme described, 1-amino-2-trifluoromethyl naphthalene via 2-trifluoromethyl-1-naphthoic acid according to Hofmann. In the Hofmann reaction, which proceeds smoothly, the forming 1-amino-2-trifluoromethyl naphthalene hydrolyzes on heating in alkaline medium. For this reason the amine had to be removed by distillation from the reaction zone already at the moment of the formation. Thus, the otherwise low yield could be increased to 61%. By this method the instability of the trifluoromethyl group in 1-amino-2-trifluoromethyl naphthalene towards aqueous alkali lyes was found (Ref 7). From the amine (VI) and  $\alpha$ -naphthylamine (Ref 8) the dyestuffs (A) were obtained by diazotization and coupling with dimethyl aniline. As may be seen from the table the introduction of the trifluoromethyl group in molecule of the dyestuff (A) shifts its absorption maximum in the direction of the short waves in neutral as well as in acid solutions. There are 1 table and 9 references, 1 of which is Soviet.

ASSOCIATION: Institut organicheskoy khimii Akademii nauk Ukrainskoy SSR  
Card 3/4 (Institute of Organic Chemistry of the Academy of Sciences

2-Trifluoromethyl Naphthalene and Its Derivatives  
of the Ukrainskaya SSR)

SOV/79-29-9-60/76

SUBMITTED: July 18, 1958

Card 4/4

5(3)

SOV/79-29-9-61/76

AUTHORS: Yagupol'skiy, L. M., Shtepanek, A. S.

TITLE: Synthesis of the Nitro Derivatives of  $\omega$ -Chlorostyrene

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 9, pp 3086-3092 (USSR)

ABSTRACT: The authors tried to use the easily accessible p- and m-nitro-phenyl chloromethyl carbinols (Refs 1, 2) for the synthesis of the corresponding nitro derivatives of  $\omega$ -chlorostyrene by substituting the chlorine for hydroxyl with a subsequent separation of HCl according to scheme (A). In the attempt of substituting chlorine in compound (I) for hydroxyl with the aid of thionyl chloride the already known compound (V) was obtained instead of the expected compound (II). In the action of  $\text{PCl}_5$  on (I) compound (II) smoothly forms, which reacts with triethylamine in alcohol according to scheme (B), and which leads to compound (IV). The structure of compound (IV) was proved according to scheme 2. m-Nitrophenyl chloromethyl carbinol (VI) which together with thionyl chloride, as mentioned above, gives compound (VII) reacts in a similar way as the para-derivative.  $\text{PCl}_5$  transforms compound (VI) into the dichloride (VIII). In the separation of HCl by an alcoholic

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SOV/79-29-9-61/76

Synthesis of the Nitro Derivatives of  $\omega$ -Chlorostyrene

solution of triethylamine an uncrystallizable oil results. In contrast to it the earlier described (Ref 5)  $\omega$ -nitro- $m$ -chlorostyrene is a solid compound. Similar to the para-isomer the preparation obtained must have the structure of  $m$ -nitro- $\alpha$ -chlorostyrene (IX). Also the hitherto undescribed 3,5-dinitro- (X) and 2,4-dinitro phenyl chloromethyl carbinol (XI) was synthesized by nitration of nitric acid ester of  $m$ - and  $p$ -nitrophenyl chloromethyl carbinol with subsequent saponification into strongly acid medium in the presence of urea. From carbinol (X) the dichloride (XII) was then obtained with  $PCl_5$ , which in the separation of  $HCl$  yielded 3,5-dinitro- $\alpha$ -chlorostyrene (XIII). The attempt of obtaining  $p$ -nitro- $\omega$ -chlorostyrene (III) by separating water from  $p$ -nitrophenyl chloromethyl carbinol could be carried out best, in order to obtain better yields, by heating carbinol (I) with phosphoric acid anhydride at  $100^\circ$ . The styrene (III) yield was 47% (besides its racemate (XIV) (4%)). By the same method the styrenes (XV) and (XVI) resulted by separating water from the corresponding carbinols. Compound (XVII) was formed as side product of the latter. It was shown that, in contrast to the meta-isomer,  $p$ -nitro- $\omega$ -chlorostyrene has a mobile

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SOV/79-29-9-61/76

Synthesis of the Nitro Derivatives of  $\omega$ -Chlorostyrene

halogen which made it possible to synthesize compounds (XVIII) - (XX). There are 7 references, 4 of which are Soviet.

ASSOCIATION: Institut organicheskoy khimii Akademii nauk Ukrainskoy SSR  
(Institute of Organic Chemistry of the Academy of Sciences  
of the Ukrainskaya SSR)

SUBMITTED: July 18, 1958

Card 3/3

VISHNEVSKAYA, G.O.; GORBUNOVA, A.S.; ZHELOBENKO, V.A.; FIALKOV, Yu.A.;  
SHEVCHENKO, O.I.; YAGUPOL'SKIY, L.M.

Synthesis of the preparation bilignost. Med. prom. 14 no.9:25-30  
S '60. (MIRA 13:9)

1. Kiyevskiy khimiko-farmatsevticheskiy zavod im. M.V. Lomonosova.  
(ADIPIIC ACID)

BC761

S/079/60/030/04/54/080  
B001/B002

53610

AUTHORS:

Yagupol'skiy, L. M., Butlerovskiy, M. A., Belinskaya, R. V.,  
Ivanova, V. I.

TITLE:

m- and p-Aminophenylethyleneglycols

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol. 30, No. 4, pp. 1288-1291

TEXT: The authors converted m- and p-nitrophenylchloromethylcarbinols (Ref. 1) which are now easily available, into m- and p-aminophenylethyleneglycols which may serve as initial substances for the synthesis of dyes and highly molecular compounds. The synthesis of m- and p-nitrophenylethyleneglycols was caused by heating of the corresponding nitrophenylchloromethylcarbinols with potassium carbonate dissolved in water:

$\text{NO}_2\text{C}_6\text{H}_4\text{CHOHCH}_2\text{Cl} \longrightarrow \text{NO}_2\text{C}_6\text{H}_4\text{CHOHCH}_2\text{OH}$ . The two glycols which are easily soluble in water, were extracted with ether or dichloroethane. m-nitrophenylethyleneglycol was also obtained by saponification of diacetyl derivative (II), according to Scheme 2. After heating with 1% sulfuric acid, the oxide of p-nitrostyrene yields p-nitrophenylethyleneglycol. The hydration process of the oxide of m-nitrostyrene is much more complicated,

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S/079/60/030/04/54/080  
B001/B002

## m- and p-Aminophenylethyleneglycols

since many by-products develop which inhibit the separation of m-nitrophenylethyleneglycol. During the oxidation of p-nitrophenylethyleneglycol with diluted nitric acid, p-nitrobenzoylcarbinol (III) develops which melts at 134-135°:  $p\text{-NO}_2\text{C}_6\text{H}_4\text{CHOHCH}_2\text{OH} \longrightarrow p\text{-NO}_2\text{C}_6\text{H}_4\text{COCH}_2\text{OH}$  (III). The authors of the present paper, in a similar way as other scientists by other methods (Refs. 2,3), obtained the acetyl derivative of carbinol (III) from p-nitrochloroacetophenone with the melting point also at 121-122°. The compound obtained by Engler and Zielke thus does not correspond to product (III) whose melting point is 132-133°C, but to the acetyl derivative. In the presence of a platinum catalyst, m- and p-nitrophenylethyleneglycol was reduced into amino compounds. High-melting products of unknown structure developed by the reduction of p-nitrophenylchloromethylcarbinol. Azo dyes were obtained from all amino compounds, by coupling with  $\beta$ -oxynaphthoic acid. There are 3 references, 1 of which is Soviet.

ASSOCIATION: Institut organicheskoy khimii Akademii nauk Ukrainskoy SSR  
(Institute of Organic Chemistry of the Academy of Sciences,  
Ukrainskaya SSR)

Card 2/3

m- and p-Aminophenylethyleneglycols

80761

S/079/60/030/04/54/080  
B001/B002

SUBMITTED: March 12, 1959

Card 3/3

20752

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8/079/60/030/04/55/080  
B001/B002

AUTHORS: Yagupol'skiy, L. M., Fialkov, Yu. A.

TITLE: 1-Phenyl-2-trifluoromethylethylene Phenyltrifluoromethyl-  
acetylene and Their Derivatives

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol. 30, No. 4, pp. 1291-1294

TEXT: The authors synthesized the vinylene homolog of benzotrifluoride (I) and the corresponding acetylene compound (II):  $C_6H_5CH=CH-CF_3$  (I),

$C_6H_5C \equiv C-CF_3$  (II). The initial product used was 1-phenyl-2-trichloro-  
methylethylene (III) which was obtained by adding trichlorobromo methane  
to styrene, and separating hydrogen bromide (Refs. 1,2). Most successful  
was the substitution of chlorine by fluorine in compound (III) by means of  
antimony trifluoride in dioxane, by which (I) was obtained in good yields.  
1-phenyl-2-trifluoromethylethylene (I) easily forms addition compounds with  
chlorine and bromine, and develops two diastereoisomers which cannot be  
dissolved by vacuum distillation. Compound (I) does not enter into the  
dienesynthesis, and according to Prilezhayev it does not develop an oxide

Card 1/3

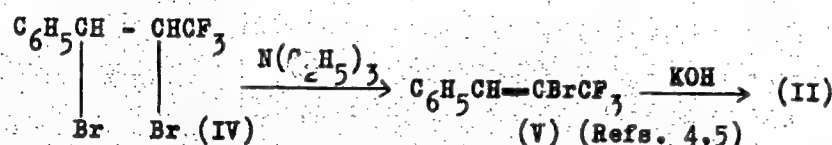


00762

1-Phenyl-2-trifluoromethylethylene, Phenyltrifluoromethylacetylene and Their Derivatives

8/079/60/030/04/55/080  
B001/B002

if benzene hydroperoxide is added. Compound (II) was synthesized according to the following scheme:



In phenyltrifluoromethylacetylene the trifluoromethyl group is stable against alkali liquor, since good yields of compound (II) are obtained from (V) by action of melted KOH at 230°. Phenyltrifluoromethylacetylene easily forms addition compounds with 2 bromine atoms; further additions do not take place. Four atoms of chlorine form an addition compound with (II). Unlike bis-(trifluoromethyl)-acetylene (Ref. 6), (II) does not form addition compounds with acetic acid. All liquid compounds obtained are described in the table. The absorption maxima and extinctions of (I) and (II) in alcohol were determined. A similar shift of the absorption maximum towards shorter waves was found in the comparison between ultraviolet spectra of styrene and phenylacetylene (Ref. 9). There are 1 table and 9 references, 1 of which is Soviet.

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1-Phenyl-2-trifluoromethylethylene, Phenyltri-  
fluoromethylacetylene and Their Derivatives

30762

S/079/60/030/04/55/080  
B001/B002

ASSOCIATION: Institut organicheskoy khimii Akademii nauk Ukrainskoy SSR ✓  
(Institute of Organic Chemistry of the Academy of Sciences,  
Ukrainskaya SSR)

SUBMITTED: March 17, 1959

Card 3/3

S/079/60/030/04/56/080  
B001/B011

AUTHORS:

Yagupol'skiy, L. M., Yufa, P. A.

TITLE:

Phenyl-bis-(trichloromethyl)-phosphinoxide, Phenyl Trichloromethyl Phosphinic Acid, and Their Derivatives

PERIODICAL:

Zhurnal obshchey khimii, 1960, Vol. 30, No. 4, pp. 1294-1299

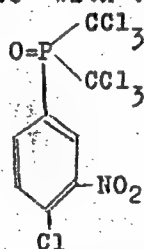
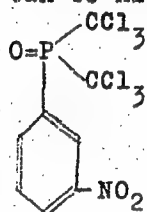
TEXT: The authors aimed at synthesizing compounds containing a phosphorus atom linked with the benzene ring and with one or two trichloromethyl groups. The oxide of  $\omega, \omega'$ -dichlorodimethyl phenyl phosphine (Ref. 1), which was chlorinated at 150-215°, served as the initial product. The resulting product was the oxide of phenyl-bis-(trichloromethyl)-phosphine (I)

$\text{C}_6\text{H}_5\text{P}(\text{CH}_2\text{Cl})_2$   $\xrightarrow{\text{O}}$   $\text{C}_6\text{H}_5\text{P}(\text{CCl}_3)_2$  (I) The oxide of  $\omega, \omega'$ -dichloro dimethyl-(p-chlorophenyl)-phosphine, which was obtained from p-chlorophenyl tetrachlorophosphorus and diazomethane, was chlorinated, and the oxide of p-chlorophenyl-

Card 1/3

Phenyl-bis-(trichloromethyl)-phosphin oxide, Phenyl S/C79/60/030/04/56/080  
Trichloromethyl Phosphinic Acid, and Their B001/B011  
Derivatives

bis-(trichloromethyl)-phosphine (II) was obtained. Both oxides (I) and (II) are colorless crystalline products and do not change on the action of aqueous acid- and alkali solutions up to 100°. They are so stable that they can be nitrated at 100° with the nitration mixture;



The ethyl ester of phenyl trichloromethyl phosphinic acid was taken as the initial product of the synthesis of the derivatives of the latter (Ref. 2). Investigations were extended to the reaction of ester (III) with  $\text{PCl}_5$ , with the acid chloride (IV) forming according to Scheme 2. In addition to the

Card 2/3

Phenyl-bis-(trichloromethyl)-phosphin oxide,  
Phenyl Trichloromethyl Phosphinic Acid, and  
Their Derivatives

S/079/60/030/04/56/080  
B001/B011

acid chloride (IV) there arises a certain amount of (V), according to Scheme 3. On heating the ester (III) with 3 moles of  $PCl_5$  at 100-160°C, a complex (VI) is formed (Scheme 4), which, on hydrolyzing, gives rise to the acid chloride (IV) in a quantitative yield. The authors became acquainted with the article by P. Biddle, I. Kennedy, I. Willans (Ref. 3) only after having completed their own investigation (Scheme 5). A paper by G. Kamay is mentioned (Ref. 2). There are 4 references, 2 of which are Soviet.

ASSOCIATION: Institut organicheskoy khimii Akademii nauk Ukrainkoy SSR  
(Institute of Organic Chemistry of the Academy of Sciences,  
Ukrainskaya SSR)

SUBMITTED: May 5, 1959

Card 3/3

YAGUPOL'SKIY, L.M.; BELINSKAYA, R.V.

Esters of acetone cyanohydrin and of aromatic acids. Zhur.ob.  
khim. 30 no.6:2014-2016 Je '60. (MIRA 13:6)

1. Institut organicheskoy khimii Akademii nauk Ukrainskoy SSR.  
(Lactonitrile) (Benzoic acid) (Benzenesulfonic acid)



YAGUPOL'SKIY, L.M.; TROITSKAYA, V.I.

Fluorine-containing analogs of anisaldehyde and piperonal. Zhur.  
ob. khim. 30 no.9:3129-3132 S '60. (MIRA 13:9)  
(Anisaldehyde) (Piperonal)

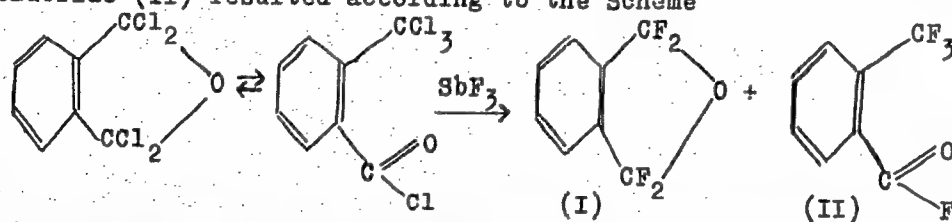
S/079/60/030/010/027/030  
B001/B066

AUTHOR: Yagupol'skiy, L. M.

TITLE: 1,1,3,3-Tetrafluoro Phthalane<sup>1</sup> and Its Derivatives

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol. 30, No. 10,  
pp. 3444 - 3448

TEXT: Considering the papers of Refs. 1-5, the author wanted to synthesize 1,1,3,3-tetrafluoro phthalane from 1,1,3,3-tetrachloro phthalane by means of antimony trifluoride. In this synthesis, however, a mixture of 1,1,3,3-tetrafluoro phthalane (I) and o-trifluoro methyl-benzoyl fluoride (II) resulted according to the Scheme



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## 1,1,3,3-Tetrafluoro Phthalane and Its Derivatives

S/079/60/030/010/027/030  
B001/B066

Compound (II) was converted into the amide by passing ammonia through the solution of the resultant mixture of (I) and (II) in petroleum ether. This amide was filtered off. 1,1,3,3-tetrafluoro phthalane, a colorless liquid melting at 153°C, was distilled off. The question as to whether the acid fluoride (II) is formed by isomerization of 1,1,3,3-tetrafluoro phthalane or by the good isomerization of 1,1,3,3-tetrachloro phthalane to the acid chloride of o-trichloro methyl benzoic acid with subsequent fluorination was examined. According to the experiments, the latter assumption is correct. It was thus shown that, unlike the corresponding chloro derivatives, 1,1,3,3-tetrafluoro phthalane and o-trifluoro methyl-benzoyl fluoride are not capable of mutual isomerization. The author also describes the synthesis of the 5-nitro-, 5-amino-, 5-cyano-, 5-carboxy-, and 5-fluoro derivatives of 1,1,3,3-tetrafluoro phthalane. The absorption spectra of all preparations are shown in three Diagrams. 1,1,3,3-tetrafluoro phthalane is stable to alkaline reagents. 6-nitro-1,1,3,3-tetrafluoro phthalane could be obtained only from 6-nitro phthalide. With dimethyl aniline, the resultant amine gives an azo dye (Scheme 2). There are 3 figures and 8 references: 2 Soviet, 2 US, and 4 German.

Card 2/3

1,1,3,3-Tetrafluoro Phthalane and Its  
Derivatives

S/079/60/030/010/027/030  
B001/B066

ASSOCIATION: Institut organicheskoy khimii Akademii nauk  
Ukrainskoy SSR (Institute of Organic Chemistry of the  
Academy of Sciences Ukrainskaya SSR)

SUBMITTED: October 30, 1959

Card 3/3

87531

53630

S/079/60/030/012/015/027  
B001/B06A

AUTHORS: Yagupol'skiy, L. M. and Ivanova, Zh. M.

TITLE: p-Trifluoro Methyl Phenyl Tetrafluoro Phosphorus and Its  
Derivatives

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol. 30, No. 12, pp.4026-4030

TEXT: The authors continued their investigation (Refs.1,2) on the synthesis of some aryl tetraphosphorus fluoride compounds, difluorides of aryl- and aryl thiophosphinic acids, and tried to synthesize the phenyl tetraphosphorus fluoride compound with an electronegative trifluoro methyl group as substituent in the molecule, and to test its stability toward hydrolysis. For this purpose, p-tolyl dichloro phosphine was produced, converted at a low-temperature by chlorination into p-tolyl tetraphosphorus dichloride, and then in the presence of azoisobutyric acid dinitrile chlorinated in the methyl group. Chlorination did, however, not proceed smoothly under various reaction conditions so that no pure products were obtained. Also the fluorination of commercial p-trichloro methyl-phenyl phosphorus tetrachloride and the dichloride of

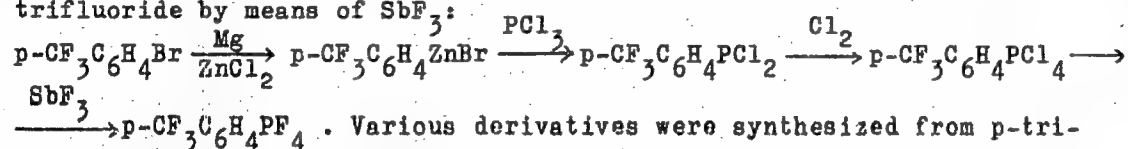
Card 1/3

87531

p-Trifluoro Methyl Phenyl Tetrafluoro  
Phosphorus and Its Derivatives

S/079/60/030/012/015/027  
B001/B064

p-trichloro methyl-phenyl phosphorus tetrachloride, as well as the dichloride of p-trichloro methyl-phenyl phosphinic acid with  $\text{SbF}_3$  or  $\text{ZnF}_2$  proved unsuccessful. Therefore, the authors proceeded from a compound already containing a trifluoro methyl group when synthesizing p-trifluoro methyl-phenyl phosphorus tetrachloride. The authors proceeded from p-bromobenzotrifluoride, which was converted into organo-magnesium and, moreover, into organo-zinc compounds, and by means of  $\text{PCl}_3$  into p-trifluoro methyl-phenyl phosphine dichloride. The latter was chlorinated and furthermore fluorinated to p-trifluoro methyl-phenyl tetraphosphorus trifluoride by means of  $\text{SbF}_3$ :



. Various derivatives were synthesized from p-trifluoro methyl-phenyl phosphorus tetrachloride. In one of previous papers (Ref.3) it was shown that phenyl phosphorus tetrachloride reacts with diazomethane by the scheme  $\text{C}_6\text{H}_5\text{PCl}_4 + 2\text{CH}_2\text{N}_2 \longrightarrow \text{C}_6\text{H}_5\text{PCl}_2(\text{CH}_2\text{Cl})_2$ .

Card 2/3

87531

p-Trifluoro Methyl Phenyl Tetrafluoro  
Phosphorus and Its Derivatives

S/079/60/030/012/015/027  
B001/B064

Phenyl phosphorus tetrachloride reacts with diazomethane less vigorously. After the reaction products had been hydrolyzed, a crystalline acid

product was separated which was identified as  $C_6H_5P(=O)(OH)CH_2F$ , i.e., phenyl- $\omega$ -

fluoro methyl phosphinic acid. In contrast to published data it was shown that phenyl phosphorus tetra-fluoride reacts immediately with carboxylic acids, under the formation of acid fluorides. Thus, phenyl phosphorus tetrafluoride gives propionic acid fluoride with propionic acid. In order to protect the glass against HF, however, a salt of this acid (e.g., the lithium salt) was used. There are 7 references: 4 Soviet, 1 US, and 2 Swiss.

ASSOCIATION: Institut organicheskoy khimii Akademii nauk Ukrainskoy SSR  
(Institute of Organic Chemistry of the Academy of Sciences  
Ukrainskaya SSR)

SUBMITTED: January 23, 1960

Card 3/3

YAGUPOL'SKIY, L.M.; KRASOVITSKIY, B.M.; BLINOV, V.A.; SIDNEVA, K.M.;  
~~PRIYASLOVA, D.G.~~

Properties of some fluorine-containing azo dyes. Zhur.prikl.  
khim. 33 no.7:389-392 J1 '60. (MIRA 13:7)

1. Institut organicheskoy khimii AN USSR. Khar'kovskiy  
gosudarstvennyy universitet. Nauchno-issledovatel'skiy  
institut organicheskikh poluproduktov i krasiteley.  
(Azo dyes)



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S/020/60/134/006/021/031  
B016/B067

AUTHORS: Yagupol'skiy, L. M. and Yagupol'skaya, L. N.

TITLE: Electron Nature of the Fluorine-containing Substituents

PERIODICAL: Doklady Akademii nauk SSSR, 1960, Vol. 134, No. 6,  
pp. 1381-1383

TEXT: The authors found that the physicochemical properties of several aromatic fluorine compounds (except for those substituted with fluorine atoms, and for trifluoro methyl derivatives of benzene) have not been investigated. They first determined the  $pK_a$  of the corresponding benzoic acids. For this purpose they measured the pH of the aqueous-alcoholic solutions half-neutralized with titrated NaOH solution. The pH was measured by means of a tube potentiometer  $\text{MP-5}$  (LP-5) with glass electrode at  $25^\circ\text{C}$ . A saturated calomel electrode served as comparison electrode. Table 1 gives the results of the determination. To determine the constant  $\rho$  of the equation by L. P. Hammett (Ref. 2)  $\log(k/k_0) = \rho \sigma$  the authors chose 5 substituents with exactly determined  $\sigma$ -constants and  $pK_a$  of the corresponding benzoic acids in aqueous ethanol. Besides

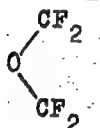
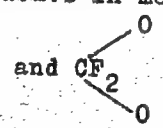
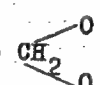
Card 1/3

B1675

Electron Nature of the Fluorine-containing  
Substituents

S/020/60/134/006/021/031  
B016/B067

they used the data for  $pK_a$  and  $\sigma = 0$  of the non-substituted benzoic acids. For calculating  $\rho$  only the  $pK_a$  of the m-derivatives of benzoic acid were used. On the basis of these data and according to the method described in Ref. 3 it was found that  $\rho = 1.535$ ; the correlation coefficient  $r$  and the standard error  $s$  were also calculated. The substituents chosen by the authors satisfy R. W. Taft's conditions (Ref. 4). They were: H, m-Br,  $CH_3CO$ ,  $CF_3$ , CH,  $SO_2CH_3$ , since in this case  $\rho = 1.535$ ,  $r = 0.998$ , and  $s = 0.03$ . On the basis of  $\rho$  the  $\sigma$  constants of the new substituents in meta- and para-position were calculated. The cyclic groups

 and  were ascribed to the p-series on the basis of the analogy with group  as well as with tetralin and hydrindene

(Refs. 2,3). All substituents mentioned are electron acceptors or very weak electron donors. The dissociation constants of the acids mentioned are higher than those of benzoic acid, the values of the  $\sigma$  constants

Card 2/3

Electron Nature of the Fluorine-containing  
Substituents

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S/020/60/134/006/021/031  
B016/B067

being positive. It appears from Table 1 that the trifluoro methyl sulfonyl group is the strongest electron-acceptor group among the known substituents in the benzene ring. p-trifluoro methyl sulfonyl benzoic acid is more than twice as strong as p-nitrobenzoic acid. This holds also for meta acids. Fig. 1 shows the dependence between the values of the  $\sigma$  constants and log k of substituted benzoic acids in 50% aqueous ethanol. The authors compared the intensity and the orientating effect of some groups and they describe the positions in which individual compounds can be nitrated. There are 1 figure, 1 table, and 8 non-Soviet references.

ASSOCIATION: Institut organicheskoy khimii Akademii nauk USSR (Institute of Organic Chemistry of the Academy of Sciences, UkrSSR)

PRESENTED: June 1, 1960, by V. N. Kondrat'yev, Academician

SUBMITTED: May 25, 1960

Card 3/3

S/020/60/135/002/029/036  
B004/B056

AUTHORS: Yagupol'skiy, L. M., Bystrov, V. F., and Utyanskaya, E. Z.

TITLE: Investigation of the Chemical Shift of the Magnetic Resonance of Fluorine<sup>19</sup> Nuclei in Fluorobenzenes With Fluorine-containing Substituents

PERIODICAL: Doklady Akademii nauk SSSR, 1960, Vol. 135, No. 2, pp. 377-380

TEXT: It was the purpose of the present work to investigate the effect of fluorine-containing substituents upon the electron density distribution in the benzene ring, and to determine the interrelation between the structure of these compounds and their reactivity by means of nuclear magnetic resonance (nmr). The shift in the ring of monosubstituted fluorobenzenes caused by fluorine and referred to the resonance values of fluorine in nonsubstituted fluorobenzene was measured and determined from the equation:

$$\delta = (H_X - H_{C_6H_5F}) \cdot 10^6 / H_{C_6H_5F} \cdot H_X \text{ and } H_{C_6H_5F} \text{ are resonance values of the}$$

Card 1/5

Investigation of the Chemical Shift of the  
Magnetic Resonance of Fluorine<sup>19</sup> Nuclei in  
Fluorobenzenes With Fluorine-containing  
Substituents

S/020/60/135/002/029/036  
B004/B056

external magnetic field for F<sup>19</sup> nuclei in the case of substituted (X) and nonsubstituted fluorobenzene. Apparatus and method of measurement are described in Ref. 1. The values for  $\delta$  are given in Table 1 for various substituents in o-, m- and p-position. The authors discuss the data given in publications for the interrelation between  $\delta$  and the Hammett constant  $\sigma$ , they compare the values obtained from various calculations, and found that substituents with positive conjugation effect  $\sigma_c$  cause a meta-position, such with negative substitution effect, however, an ortho- or para-position. Tables 3 and 4 compare the values of  $\delta_p$ ,  $\sigma_p$  for the p-position and of  $\delta_m$ ,  $\sigma_m$  for meta-position. Besides, Table 3 gives the light absorption  $\lambda_{max}$  of 4'-substituted dimethylaminoazobenzene, which changes similar to the chemical shift in p-substituted fluorobenzenes, and indicates the existence of uniform electron displacements. There are 4 tables and 9 references: 3 Soviet, 7 US, and 1 French.

Card 2/5

Investigation of the Chemical Shift of the  
Magnetic Resonance of Fluorine<sup>19</sup> Nuclei in  
Fluorobenzenes With Fluorine-containing  
Substituents

S/020/60/135/002/029/036  
E004/B056

ASSOCIATION: Institut organicheskoy khimii Akademii nauk USSR (Institute  
of Organic Chemistry of the Academy of Sciences USSR).  
Institut khimicheskoy fiziki Akademii nauk SSSR (Institute  
of Chemical Physics of the Academy of Sciences USSR)

PRESENTED: June 1, 1960 by V. N. Kondrat'yev, Academician

SUBMITTED: May 25, 1960

Card 3/5

S/020/60/135/002/029/036  
B004/B056

Химические сдвиги фтора  
в бензольном кольце 1)

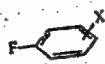


Table 1

Заместитель X 2)	o	m	p	3,4)
SO <sub>2</sub> CF <sub>3</sub>	-8,7	-4,7	-15,1	
SCF <sub>3</sub>	-7,8	-2,3	-4,7	
OCF <sub>3</sub>	+17,4	-2,3	+2,8	
CH=CH-CF <sub>3</sub>			-2,4	
				+3,9
				-8,6

Legend to Table 1: 1) Chemical shifts of fluorine in the benzene ring; 2) substituent

Card 4/5

SO <sub>2</sub> , CH=CH-CF <sub>3</sub>	J	Br	Cl	OCF <sub>3</sub> , CF <sub>3</sub> , O	P	SCF <sub>3</sub>	CH <sub>3</sub>	OCF <sub>3</sub>
-1,7	1,2	2,3	2,4	3,9	6,4	—	5,5	11,4
0,38	0,23	0,23	0,23	0,36	0,06	—	—	-0,17
0,32	—	420	420	420	—	—	408	407

Таблица 4

P	Cl	Br	J	SCF <sub>3</sub>	OCF <sub>3</sub>	SCF <sub>3</sub>	OCF <sub>3</sub>	CH <sub>3</sub>
-3,1	-2,1	-2,4	-2,6	-2,3	-2,3	—	-1,3	+0,9
0,34	0,37	0,39	0,35	0,35	0,36	0,15	0,12	-0,07

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S/020/60/135/002/029/000  
B004/B056

Legend to Tables 3 and 4  
1) Substituent,  
2) (in alcohol)

Таблица 4								
Заместитель 1)		SO <sub>2</sub> CF <sub>3</sub>	NO <sub>2</sub>	SO <sub>2</sub> CH <sub>3</sub>	CN	CF <sub>3</sub> CF <sub>3</sub> O	COCH <sub>3</sub>	CF <sub>3</sub>
$\delta_H$		-15,1	-10,8	—	-9,6	-8,6	-8,6	-5,1
$\sigma_H$		1,03	0,78	0,72	0,66	0,81	0,50	0,54
$\lambda_{max}$ (I), мμ (в спирте)		476	475	445	—	450	447	430
	2)							

Заместитель 1)		SO <sub>2</sub> CF <sub>3</sub>	NO <sub>2</sub>	SO <sub>2</sub> CH <sub>3</sub>	CN	CF <sub>3</sub>
$\delta_M$		-4,7	-3,3	—	-3,0	-2,8
$\sigma_M$		0,70	0,71	0,65	0,56	0,43



YAGUPOL'SKIY, L.M.; GRUZ, B.Ye.; MAN'KO, N.I.; KIPRIANOV, A.I.

Synthesis of bilitrast-- $\beta$ -(4-hydroxy-3,5-diiodophenyl)- $\alpha$ -phenyl-  
propionic acid. Ukr. khim. zhur. 226 no.2:233-236 '60.  
(MIRA 13:9)

1. Institut organicheskoy khimii AN USSR.  
(Phloretic acid)

BYSTROV, V.F.; UTYANSKAYA, E.Z.; YAGUPOL'SKIY, L.M.

Magnetic resonance spectra of  $^{19}\text{F}$  in aromatic compounds with  
fluorine-containing substituents. Opt. i spektr. 10 no. 1:138-  
141 Ja '61. (MIRA 14:1)

(Fluorine--Spectra)

YAGUPOL'SKIY, L.M.; D'YACHENKO, Ye.P.; TROITSKAYA, V.I.

*p*-Trichloromethylmercapto- and *p*-trichloromethoxybenzoic acids  
and their derivatives. Ukr. khim. zhur. 27 no. 1:77-79 '61.  
(MIRA 14:2)

1. Institut organicheskoy khimii AN USSR.  
(Anisic acid) (Benzoic acid)

YAGUPOL'SKIY, L.M.; BELINSKAYA, R.V.

Alkylation with esters of fluorine-containing carboxylic acids.  
Zhur. ob. khim. 31, no.1:336-337 Ja '61. (MIRA 14:1)

1. Institut organicheskoy khimii Akademii nauk Ukrainskoy SSR.  
(Alkylation) (Esters)

YAGUPOL'SKIY, L.M.; TAOITSKAYA, V.I.

Cyanine dyes containing fluoroine. Part 9: Cyanine dyes from derivatives of 5,6-(Difluoromethylene)-dioxymethythiazole.  
Zhur. ob. khim. 31 no. 2:628-632 F '61. (MIRA 14:2)

1. Institut organicheskoy khimii AN USSR.  
(Cyanine dyes) (Benzothiazole)

YAGUPOL'SKIY, L.M.; TROITSKAYA, V.I.

Synthesis of derivatives of phenyl triflouromethyl ether.  
Zhur. ob. khim. 31 no.3:915-924 Mr '61. (MIRA 14:3)

1. Institut organicheskoy khimii AN USSR.  
(Ether)

YAGUPOL'SKIY, L.M.; GRUZ, B. Ye.

Synthesis of some derivatives of phenyl trifluoromethyl sulfide and phenyl trifluoromethyl sulfone. Zhur. ob. khim. 31 (MIRA 14:4)  
no.4:1315-1320 Ap '69. 61

1. Institut organicheskoy khimii Akademii nauk Ukrainskoy SSR.

(Sulfide) (Sulfone)

30186

S/079/61/031/011/008/015  
D202/D305

53600

AUTHORS:

Yagupol'skiy, L. M., and Fialkov, Yu. A.

TITLE:

Derivatives of the vinyl homologue of benzo-tri-fluoride

PERIODICAL:

Zhurnal obshchey khimii, v. 31, no. 11, 1961, 3586-3593

TEXT:

This is a continuation of previous work in which a new compound 1-phenyl-2-trifluoro methylene has been found (cpd. I). In this investigation, the author's aim was to obtain the para and meta derivatives of the above compound. n-Bromo-styrene gave an addition product with  $\text{CBrCl}_3$  (in the presence of benzoylperoxide), forming n-bromo-phenyl trichlorobromopropane:  $\text{Br}-\text{C}_6\text{H}_4-\underset{\text{Br}}{\text{CH}}-\text{CH}_2-\text{CCl}_3$ ; a crystalline product,

m.p. 84.5 - 85.5; after the removal of  $\text{HBr}$ , n-bromo-phenyl trichloromethylene was obtained. (Cpd. II), an oily substance, b.p. 120 - 121°C

Card 1/5



30186

S/079/61/031/011/006/015  
D202/D305

Derivatives of the...

(0.1 mm) : By the action of  $\text{SbF}_3$  on cpd. II it was converted to n-bromo-phenyl-trifluoro-methylene (cpd. III) crystals, m.p.  $21.5 - 22.5$ , b.p.  $115^\circ$  (25 mm). This compound heated with  $\text{CuCN}$  formed (in pyridine) n-cyano-phenyl-trifluoromethylene (cpd. IV) crystals, m.p.  $48 - 49.5^\circ$ . Compound IV was oxidized with  $\text{H}_2\text{O}_2$  to form n-amide (cpd. V) crystals, m.p.  $189 - 190^\circ$ . By hydrolysis of cpd. V, a n-carboxy compound was obtained (cpd. VI), a crystalline product, m.p.  $246 - 247^\circ$ . The same cpd. VI was obtained by the action of butyl Li on cpd. III and subsequent action of solid  $\text{CO}_2$ ; m.p.  $247 - 248^\circ$ . The amide V was converted into the n-amine by Hofmann's method to give:  $\text{CF}_3\text{-CH=CH-C}_6\text{H}_4\text{-NH}_2$  (cpd. VII) crystals, m.p.  $82 - 83^\circ$ , and the n-amine was converted by the action of  $\text{NaNO}_2$  and that of sodium fluo-borate into n-fluoro phenyl trifluoro methylene, b.p.  $165 - 166^\circ$ ,  $n_D^{21} 1.4625$ ,  $n_D^{21} 1.2682$  (cpd. VIII). By diazotization and steam-distillation: VII - p-trifluoro methylene phenol (IX) was obtained in crystals, m.p.  $71.5 - 72.5^\circ$ . To

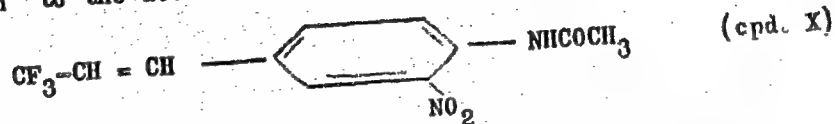
Card 2/6

30186

S/079/61/031/011/006/015  
D202/D305

Derivatives of the...

prepare meta-derivatives of cpd. I, the authors used its n-acetamide, by nitration of which a nitro compound was formed, the nitro-group in the ortho position to the acetamide:



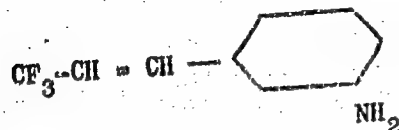
From this, by a reduction-cyclization reaction, 2-methyl-5-(trifluoro methylene)-benz-imidazol was obtained (cpd. XI). Cpd. X formed yellow needles, m.p. 169 - 170°, and cpd. XI had m.p. of 208 - 209°C. Cpd. X, after hydrolysis formed 4-amino-3-nitro phenyl trifluoro methylene; orange-red needles, m.p. 120 - 121°; this compound, after desamination gave m-nitro phenyl trifluoro methylene; a crystalline product of m.p. 42.5 - 43.5°C. From the latter, after hydration with platinum black, m-trifluoro methylene aniline was obtained:

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S/079/61/031/011/006/015  
D202/D305

Derivatives of the...



; m.p.  $29.5^\circ$ , and from it, by hydrolysis,

the corresponding phenol (cpd. XII), an oily liquid of b.p.  $86^\circ$  (0.2 mm). The authors investigated the action of alkali on the trifluoro group of the above compounds and found that this group is much more stable towards alkali than towards acid with the exception of the *n*-phenol derivative, which splits off F under the action of NaOH and gives, after prolonged boiling with the alkali, *n*-coumaric acid. The vinyl group between the benzene ring and the  $\text{-CF}_3$  group markedly diminishes its influence on the ring. There are 10 references: 6 Soviet-bloc and 4 non-Soviet-bloc. The references to the English-language publications read as follows: R. G. Jones, J. Am. Chem. Soc., 69, 2346 (1947); A. Titley, J. Chem. Soc., 2571, 1928; F. Power, A. Salway, J. Chem. Soc., 97, 236, (1910).

Card 4/5

30136

Derivatives of the...

S/079/61/031/011/006/013  
D202/D305

ASSOCIATION: Institut organicheskoy khimii akademii nauk Ukrainssou SSR  
(Institute of Organic Chemistry, AS UkrSSR)

SUBMITTED: December 12, 1960

Card 5/5

YAGUPOL'SKIY, I.M.; FIALKOV, Yu.A.; YUFA, P.A.

2-Trifluoromethylnaphthalene and its derivatives. Zhur.ob.  
khim. 31 no.12:3962-3970 D '61. (MIRA 15:2)

1. Institut organicheskoy khimii AN Ukrainskoy SSR.  
(Naphthalene)

YAGUPOL'SKIY, L.M.; GRUZ, B.Ye.

Cyanine dyes containing fluorine. Part 10: Cyanine dyes  
containing fluorine in the polymethine chain. Zhur.ob.khim.  
31 no.12:3955-3961 D '61. (MIRA 15:2)

1. Institut organicheskoy khimii AN Ukrainskoy SSR.  
(Cyanines)  
(Dyes and dyeing)  
(Fluorine)

YEGOROV, Yu.P.; LOKHMACHEV, V.F.; YAGUPOL'SKIY, L.M.

Infrared spectra of 1-phenyl-2-trifluoro ~~Methylethylene~~ and its derivatives. Izv. AN SSSR.Ser.fiz. 26 no.10:1276-1277 0 '62. (MIRA 15:10)

1. Institut khimii polimerov i monomerov AN UkrSSR.  
(Ethylene—Spectra)

YAGUPOL'SKIY, L.M.; TROITSKAYA, V.I.; MALICHENKO, B.F.

Synthesis of derivatives of diphenyl- $\alpha,\alpha$ -difluoromethane. Zhur.ob.  
khim. 32 no.6:1832-1836 Ja '62. (MIRA 15:6)  
(Methane)



GERASIMENKO, Yu.Ye.; SHEYN, S.M.; BAKULINA, G.G.; CHEREPIVSKAYA, A.P.;  
SEMENYUK, G.V.; YAGUPOL'SKIY, L.M.

Thioindigoid dyes. Part 9: Thioindigoid dyes containing fluorine.  
Zhur.ob.khim. 32 no.6:1870-1874 Je '62. (MIRA 15:6)  
(Thioindigo)

S/079/62/032/009/009/01T  
1048/1242

AUTHORS: Yagupol'skiy, L.M. and Malichenko, B.F.

TITLE: The derivatives of 1,2-diphenyl-1,1,2,2-tetrafluoroethane. I. The synthesis of 1,2-diphenyl-1,1,2,2-tetrafluoroethane-4,4'-dicarboxylic acid

PERIODICAL: Zhurnal obshchey khimii, v.32, no.9, 1962, 3035-3039

TEXT: Experimental methods for the synthesis of 1,2-diphenyl-1,1,2,2-tetrafluoroethane-4,4'-dicarboxylic acid (or its tetrachloro analog), some of its derivatives, and some intermediate products are described. The  $-CF_2-CF_2-$  group in these derivatives imparts a high resistance to hot alkaline solutions. The hydrolysis of bis(n-trichloromethyl)-benzene in the presence of ferric chloride yielded n-trichloromethylbenzoylchloride which was the starting material for most of the syntheses reported. n-trichloromethyl benzoic acid was prepared by adding triethylamine to a solution of n-trichloromethylbenzoylchloride in acetone in the presence of a small amount of water; the mixture was allowed to stand overnight and mixed with a

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I048/I242

The derivatives of 1,2-diphenyl...

10% HCl solution. The methyl ester of this acid was prepared by adding methanol and triethylamine to a solution of n-trichloromethylbenzoylchloride in benzene. The reaction of n-trichloromethylbenzoic acid dissolved in pyridine and Cu powder yielded 1,2-diphenyl-1,1,2,2-tetrachloroethane-4,4'-dicarboxylic acid, which, on boiling with thionyl chloride, yielded the dichloroanhydride. A mixture of this dichloroanhydride with antimony pentachloride and anhydrous HF was heated in an autoclave to 140-165°C yielding the analogous difluoroanhydride which on hydrolysis gave 1,2-diphenyl-1,1,2,2-tetrafluoroethane-4,4'-dicarboxylic acid. The synthesis of the following compounds are also described in detail: the dimethyl ester, the diamide, the diazide, and diisocyanate derivatives of both 1,2-diphenyl-1,1,2,2-tetrafluoroethane-4,4'-dicarboxylic acid and its tetrachloro analog, as well as the synthesis of 4,4'-bis(trichloromethyl)-1,2-diphenyl-1,1,2,2-tetrachloroethane.

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S/079/82/032/009/009/011  
I048/I242

The derivatives of 1,2-diphenyl...

ASSOCIATION: Institut organicheskoy khimii Akademii nauk  
Ukrainskoy SSR (The Institute of Organic Chemistry,  
Academy of Sciences UkrSSR)

SUBMITTED: September 6, 1961

Card 3/3

KULIK, V.F.; YEGOROV, Yu.P.; MARENETS, M.S.; YAGUPOL'SKIY, L.M.

Infrared spectra and polar effects in para-substituted benzene containing the groups  $\text{SCF}_3$ ,  $\text{SOCF}_3$ ,  $\text{SO}_2\text{CF}_3$ , and  $\text{OCF}_3$ . Zhur.strukt. khim. 4 no.4:541-547 J1-Ag '63. (MIRA 16:9)

1. Institut khimii polimerov i monomerov AN UkrSSR, Kiyev.  
(Benzene derivatives—Absorption spectra)

VISHNEVSKAYA, G.I.; KHASKIN, I.G.; BUTLEROVSKIY, M.A.; YAGUPOL'SKIY, L.M.;  
LITVINCHUK, O.D.; YAKOVLEVA, V.Ya.; GORBUNOVA, A.D.; KIRIYENKO, S.S.

Preparation of syntomycin by dichloroacetylation of  
1-p-nitrophenyl-2-aminoethanol. Ukr. khim. zhur. 29 no. 9: 947-950  
'63. (MIRA 17:4)

1. Institut organicheskoy khimii AN UkrSSR.

GITIS, S. S.; GLAZ, A. I.; YAGUPOL'SKIY, L. M.

Reactions of aromatic nitro compounds. Part 14: Effect of  
some electron acceptor groups on the reaction of re-esteri-  
fication. Zhur. ob. khim. 33 no.1:138-141 '63.  
(MIRA 16:1)

1. Institut organicheskoy khimii AN UkrSSR.

(Esterification) (Nitro compounds)

YAGUPOL'SKIY, L. M.; FIALKOV, Yu. A.

Saponification of nitric esters, derivatives of p-nitrophenyl-  
methylecarbinol. Zhur. ob. khim. 33 no.1:309-314 '63.  
(MIRA 16:1)

1. Institut organicheskoy khimii AN UkrSSR.

(Nitric acid) (Methanol) (Saponification)



YAGUPOL'SKIY, L.M.; KONDRATENKO, N.V.

Aryl sulfonyltrifluoromethylsulfonylmethanes. Zhur.ob.khim.  
33 no.3:920-928 Mr '63. (MIRA 16:3)

1. Institut organicheskoy khimii AN UkrSSR.  
(Sulfone) (Methane)

YAGUPOL'SKIY, L.M.; GANDEL'SMAN, L.Z.

Diazastyrils with fluorine-containing substituents. Zhur.ob.khim.  
33 no.7:2301-2307 J1 '63. (MIRA 16:8)

1. Institut organicheskoy khimii AN UkrSSR.  
(Diazo compounds) (Dyes and dyeing)

YAGUPOL'SKIY, L.M.: BELINSKAYA, R.V.

Fluorination of derivatives of 1,1,3,3-tetrachlorophthalan.  
Zhur.ob.khim. 33 no.7:2358-2364 J1 163. (MIRA 16:8)

1. Institut organicheskoy khimii AN UkrSSR.  
(Phthalan) (Fluorination)

YAGUPOL'SKIY, L.M.; VISHNEVSKAYA, G.O.; KAGANOVSKAYA, M.I.

Analogs of syntomycin containing trifluoromethyl-, mercapto-,  
and trifluoromethylsulfonyl groups. Zhur. ob. khim. 33 no.8:  
2721-2723 Ag. '63. (MIRA 16:11)

1. Institut organicheskoy khimii AN UkrSSR.

BYSTROV, V.F.; YAGUPOL'SKIY, L.M.; STEPANYANTS, A.U.; FIALKOV, Yu.A.

6 - Constants of substituents with a trifluoromethyl group.  
Dokl. AN SSSR 153 no.6:1321-1324 D '63. (MIRA 17:1)

1. Institut khimicheskoy fiziki AN SSSR. Predstavleno akademi-  
kom V.N. Kondrat'yevym.

YAGUPOL'SKIY, L.M.; KLYUSHNIK, G.I.; TROITSKAYA, V.I.

Cyanine dyes containing fluorine. Part 11: Synthesis of cyanine dyes from fluorine derivatives of 2-methylbenzimidazole. Zhur.ob.khim. 34 no.1:307-317 Ja '64. (MIRA 17:3)

1. Institut organicheskoy khimii AN UkrSSR.

YAGUPOL'SKIY, L. M.; ORDA, V. V.

Bis(trifluoromethoxy- and trifluoromethylmercapto) derivatives  
of benzene. Zhur. ob. Khim. 34 no.6:1979-1984 Je '64.  
(MIRA 17:7)

1. Institut organicheskoy khimii AN Ukrainskoy SSR.

YAGUPOL'SKIY, L.M.; PANTELEYMONOV, A.G.; ORDA, V.V.

Magnesium and lithium derivatives of trifluoromethylmethylsulfone  
and their reactions. Zhur. ob. khim. 34 no.10:3456-3462 O '64.  
(MIRA 17:11)

1. Institut organicheskoy khimii AN UkrSSR i Institut khimii polimerov  
i monomerov AN UkrSSR.



YAGUPOL'SKIY, L.M.; BYSTROV, V.F.; STEPANYANTS, A.U.; FIALKOV, Y.A.

Effect of the substituents with a trifluoromethyl group on the reactivity of aromatic compounds. Zhur. ob. khim. 34 no.11: 3682-3690 N '64 (MIRA 18:1)

1. Institut organicheskoy khimii AN UkrSSR i Institut khimicheskoy fiziki AN SSSR.

GRUZ, B. Ye.; STERN, R.J.; YAGUPOL'SKIY, L.M.

Reaction of N-eth<sup>yl</sup> orthoaminothiophenol with perfluoroacryl  
chloride. Zhur. ob. khim. 34 no.12:4122-4123 D '64

(MIRA 18:1)

1. Institut organicheskoy khimii AN UkrSSR.

Source: Bulleten' izobreteniy i tovarnykh znakov, no. 2, 1965, 86

Topic: the current photographic material, photographic emulsion, color

halide photographic materials to the green, yellow, and orange bands of the

100

where R = H or VII - groups; R' = SOCF<sub>3</sub> or SO<sub>2</sub>CF<sub>3</sub> - groups; R - alkyl or

sensitive layer in layered materials, after the introduction of sensitizers, the  
emulsion also receives sensitizing components, such as the pyrazolone deriva-

KULIK, V.F.; YEGOROV, Yu.P.; PANTELEYMONOV, A.G.; FIALKOV, Yu.A.; YAGUPOL'SKIY,  
L.M.

Electronic interaction and infrared spectra of para-derivatives of  
benzene  $X - C_6H_4 - Y - CF_3$ . Teoret. i eksper. khim. 1 no.2:171-178  
Mr-Ap '65. (MIRA 18:7)

1. Institut khimii vysokomolekulyarnykh soyedineniy AN UkrSSR,  
Kiyev i Institut organicheskoy khimii AN UkrSSR, Kiyev.

ORDA, V.V.; YAGUPOL'SKIY, L.M. [IAhupol's'kyi, L.M.]; BYSTROV, V.F.;  
STEPANYANTS A.U.

Transmission of the induction effect of  $\text{SCF}_3$  -  $\text{SOCF}_3$  and  
 $\text{SO}_2\text{CF}_3$  substituents through the methylene group. Dop. AN  
URSR no.3:345-348 '65.

(MIRA 18:3)

1. Institut organicheskoy khimii AN UkrSSR.

YAGUPOL'SKIY, L.M.; MALICHENKO, B.F.

Difluoro (and dichloro) ethylene-1,2 - diphenyl-4,4'-  
dicarboxylic acids. Zhur. ob. khim. 35 no.3:490-493 Mr '65.  
(MIRA 18:4)

1. 17/11 PNC(1)/T EFD(b)-3 Pae-2 LP(1)

8/226/65/000/005/0.03/0104

... silver halide photographic materials. Class 37,

... 1965, 101-104

AR 771. This is a certificate presents a method for sensitizing silver halide photographic materials to the green, yellow, and orange bands of the spectrum. The method involves the introduction of sensitizers such as imidazo-  
cyanine and azo dyes. The sensitizers are derivatives of the imidazo-  
cyanine and azo dyes.



sensitivity to photographic materials, imidocarboc- and imidomethanimerocyanine  
are used as sensitizers. The residues of these compounds contain trifluoromethyl-  
-2- trifluoromethyl sulfoxide groups.

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AN USSR (Institute of Organic Chemistry)

SUB CODE: CC, CC

ENCL: 00

SUBJECT: --

OTHER: 000

NO REF SCV: 000

Card 2/2

YAGUPOL'SKIY, L.M.; BELINSKAYA, R.V.

Isomerization of 1,1,3,3-tetrahalophthalans. Zhur. ob. khim.  
35 no. 6:969-977 Ju '65. (MIRA 18:6)

1. Institut organicheskoy khimii AN UkrSSR.

YACUPOL'SKIY, L.M.; FIALKOV, Yu.A.

Vinylene homolog of benzotrifluoride. Part 3: Polarization  
of a double bond in the derivatives of 1-phenyl-2-trifluoro-  
methylethylene. Zhur. ob. khim. 35 no.6:1088-1091 Je '65.  
(MIRA 18:6)

1. Institut organicheskoy khimii AN UkrSSR.

YAGUPOL'SKIY, L.M.; PANTELEYMONOV, A.G.

Trifluoromethylmethysul'oxide. Zhur. ob. khim. 35 no.6:1120  
Ja '65. (MIRA 18:6)

1. Institut organicheskoy khimii i Institut khimii vysokomole-  
kulyarnykh soyedineniy AN UkrSSR.

YAGUPOL'SKIY, L.M.; GANDEL'SMAN, L.Z.

Effect of substituents on the color of N,N-dialkylaminoazo dyes  
and their salts. Zhur. ob. khim. 35 no.7:1252-1260 J1 '65.  
(MIRA 18:8)

1. Institut organicheskoy khimii AN UkrSSR.

YAGUPOL'SKIY, L.M.; SHEYN, S.M.; KRASNOSEL'SKAYA, M.I.; SOLODUSHENKOV, S.N.

New method for the preparation of 2-amino-4-trifluoromethylbenzoic acid. Zhur. ob. khim. 35 no.7:1261-1263, J1 '65.

(MIRA 18:8)

YAGUPOL'SKIY, I.M.; MATYUSHECHEVA, G.I.

Alkylbenzotrifluorides. Part 2: Polymethylbenzotrifluorides.  
Zhur. ob. khim. 35 no.8:1422-1426 Ag '65. (MIRA 18:8)

1. Institut organicheskoy khimii AN UkrSSR.



YAGUPOL'SKIY, L.M.; MALICHENKO, B.F.

1,2-Diphenyl-1-1-2-2-tetrafluoroethane derivatives. Part 3:  
Effect of 4,4'-substituents of  $\alpha, \beta$ -difluorostilene on the  
addition of fluorine. Zhur. ob. khim. 35 no.9:1561-1564 S '65.  
(MIRA 18:10)

1. Institut organicheskoy khimii AN UkrSSR.

YACUPOL'SKIY, L.M.; TROITSKAYA, V.I.

1,2-Diphenyl-1,1,2,2-tetrafluoroethane derivatives. Part 4:  
Amino derivatives of 1,2-diphenyl-1,1,2,2-tetrafluoroethane.  
Zhur. ob. khim. 35 no.9:1612-1620 S '65. (MIRA 18:10)

1. Institut organicheskoy khimii AN UkrSSR.

ORDA, V.V.; YAGUPOL'SKIY, L.M.; BYSTROV, V.F.; STEPANYANTS, A.U.

Transmission of the induction effect of substituents  $\text{SCF}_3$ ,  $\text{SOCF}_3$ ,  
and  $\text{SO}_2\text{CF}_3$  through a methylene group. Zhur. ob. khim. 35  
no.9:1628-1636 S '65. (MIRA 18:10)

1. Institut organicheskoy khimii AN UkrSSR i Institut khimicheskoy  
fiziki AN SSSR.

GRUZ, B.Ye.; YAGUPOL'SKIY, I.M.

2-( $\beta,\beta,\beta$ -Trifluoroethyl) benzothiazole, 2-( $\alpha,\beta,\beta,\beta$ -tetrafluoroethyl) benzothiazole and their reactions. Zhur. ob. khim. 35 no.9:1639-1644 S '65. (MIRA 18:10)

1. Institut organicheskoy khimii AN UkrSSR.